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VERIFICATION OF TRANSLATION

Sir:

I, Isamu Ogoshi, having been warned that willful false statements and the like are punishable by fine or imprisonment or both, under section 1001 of Title 18 of the United States Code, and may jeopardize the validity of the above-captioned application and any patent issuing thereon, declare:

(1) I am a patent attorney authorized to practice law in Japan and am engaged in the practice of law with OGOSHI International Patent Office at Toranomon 9 Mori Bldg. 3F, 2-2, Atago 1-Chome, Minato-ku, Tokyo 105-0002, Japan.

(2) I am fluent in the Japanese and English Languages.

(3) I have reviewed the attached translation, and certify that it is an accurate English translation of the Japanese language international application of Kunihiro Oda filed on July 29, 2003 and given International Application No. PCT/JP03/09574.

(4) All of the statements made herein of my own knowledge are true and all statements made herein on information and belief are believed to be true.

April 20, 2005
Date

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Ta SPUTTERING TARGET AND METHOD FOR PREPARATION THEREOF

TECHNICAL FIELD

The present invention relates to a manufacturing method of a sputtering target in which a Ta ingot or billet formed by melting and casting is subject to forging, annealing, rolling processing and the like, and to a Ta sputtering target obtained thereby.

BACKGROUND ART

In recent years, the sputtering method for forming a film from materials such as metal or ceramics has been used in numerous fields such as electronics, corrosion resistant materials and ornaments, catalysts, as well as in the manufacture of cutting/grinding materials and abrasion resistant materials.

Although the sputtering method itself is a well-known method in the foregoing fields, recently, particularly in the electronics field, a Ta sputtering target suitable for forming films of complex shapes and forming circuits is in demand.

Generally, this Ta target is manufactured by hot forging and annealing (heat treatment) an ingot or billet formed by performing electron beam melting and casting a Ta material, and thereafter performing rolling and finishing processing (mechanical processing, polishing, etc.) thereto. Such hot forging and annealing can be repeated. In this kind of manufacturing procedure, the hot forging performed to the ingot or billet will destroy the cast structure, disperse or eliminate the pores and segregations, and, by further annealing this, recrystallization will occur, and the precision and strength of the structure can be improved.

In this kind of manufacturing method of a target, ordinarily, recrystallization annealing is performed at a temperature of roughly 1173K (900°C). An example of a conventional manufacturing method is described below.

Foremost, the tantalum raw material is subject to electronic beam melting and thereafter cast to prepare an ingot or billet, and subsequently subject to cold forging – recrystallization annealing at 1173K - cold forging – recrystallization annealing at

1173K - cold rolling – recrystallization annealing at 1173K – finish processing to form a target material. In this manufacturing process of a Ta target, the melted and cast ingot or billet generally has a crystal grain diameter of 50mm or more.

As a result of subjecting the ingot or billet to hot forging and recrystallization annealing, the cast structure is destroyed, and generally even and fine (100 μm or less) crystal grains can be obtained. Nevertheless, with the conventional forging and annealing manufacturing method, there is a problem in that a pattern in the form of wrinkles or streaks is formed from the center to the peripheral edge of the disk.

Fig. 2 is a diagram showing the schematic of the target surface, and several to several ten black patterns have appeared. The micrograph of the structure of this portion of the crystal grains is shown in Fig. 3. Although there is no significant difference in the crystal grain diameter, heterophase crystal grains gathered in the form of wrinkles were observed in a part of the ordinary structure.

Generally, upon performing sputtering, finer and more uniform the crystals of the target, more even the deposition, and a film having stable characteristics with low generation of arcing and particles can be obtained.

Therefore, the existence of irregular crystal grains in the target that are generated during forging, rolling or the annealing to be performed thereafter will change the sputtering rate, and there is a problem in that evenness (uniformity) of the film will be affected, generation of arcing and particles will be promoted, and the quality of sputtered deposition may deteriorate thereby.

Further, if a forged product with stress remaining therein is used as is, the quality will deteriorate, and this must be avoided at all costs.

Accordingly, with the conventional forging and annealing process, there is a problem in that irregular crystal grains will be generated in the Ta sputtering target, and the quality of the film will deteriorate as a result thereof.

DISCLOSURE OF THE INVENTION

The present invention was devised in order to overcome the foregoing problems, and, as a result of improving and devising the forging process and heat treatment process, the crystal grain diameter can be made fine and uniform, and a method of stably manufacturing a Ta sputtering target superior in characteristics can be obtained.

The present invention provides:

1. A manufacturing method of a Ta sputtering target in which a Ta ingot or billet formed by melting and casting is subject to forging, annealing, rolling processing and the like to prepare a sputtering target, wherein the ingot or billet is forged and thereafter subject to recrystallization annealing at a temperature of 1373K to 1673K;

2. A manufacturing method of a Ta sputtering target according to paragraph 1 above, wherein forging and recrystallization annealing at a temperature of 1373K to 1673K are repeated at least twice;

3. A manufacturing method of a Ta sputtering target according to paragraph 1 or paragraph 2 above, wherein the recrystallization annealing after the forging or rolling conducted in the recrystallization annealing at a temperature of 1373K to 1673K is performed at a temperature between the recrystallization starting temperature and 1373K;

4. A manufacturing method of a Ta sputtering target according to any one of paragraphs 1 to 3 above, wherein, after the final rolling processing, recrystallization annealing is performed at a temperature between the recrystallization starting temperature and 1373K, and finish processing is further performed to obtain a target shape;

5. A manufacturing method of a Ta sputtering target according to paragraph 4 above, wherein, after performing rolling, crystal homogenization annealing or stress relieving annealing is performed;

6. A manufacturing method of a Ta sputtering target according to any one of paragraphs 1 to 5 above, wherein the average crystal grain diameter of the target is made to be a fine crystal grain size at 80 μm or less;

7. A manufacturing method of a Ta sputtering target according to any one of paragraphs 1 to 5 above, wherein the average crystal grain diameter of the target is made to be a fine crystal grain size at 30 to 60 μm; and

8. A manufacturing method of a Ta sputtering target according to any one of paragraphs 1 to 7 above, and a Ta sputtering method obtained with said method, wherein there is no uneven macro structure in the form of streaks or aggregates on the surface or inside the target.

BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is a micrograph showing the structure of the Ta target obtained by performing the forging and recrystallization annealing of the present invention. Fig. 2 is a photograph showing the schematic structure of the Ta target obtained by performing conventional forging and recrystallization annealing. Fig. 3 is a micrograph showing the structure of the Ta target obtained by performing conventional forging and recrystallization annealing.

BEST MODE FOR CARRYING OUT THE INVENTION

The sputtering target of the present invention is manufactured with the following process. To exemplify a specific example, foremost, a tantalum raw material (usually, high purity Ta of 4N5N or more is used) is melted via electronic beam melting or the like, and this is cast to prepare an ingot or billet. Next, this ingot or billet is subject to a series of processing steps including cold forging, rolling, annealing (heat treatment), finish processing and so on. Although this manufacturing process is basically the same as the conventional technology, what is particularly important is that recrystallization annealing (heat treatment) be performed at a temperature of 1373K to 1673K.

The forging performed to the ingot or billet will destroy the cast structure, disperse or eliminate the pores and segregations, and, by further annealing this, recrystallization will occur, and the precision and strength of the structure can be improved by this cold forging and recrystallization annealing. Further, when recrystallization annealing is specifically performed at a high temperature of 1373K to 1673K, it is possible to completely eliminate the pattern in the form of streaks that appeared in conventional technology.

Therefore, as shown in the micrograph of the crystal grain structure on the target surface illustrated in Fig. 1, heterophase crystal grains gathered in the form of wrinkles could not be observed in the peripheral standard crystal structure, and a uniform target structure was obtained.

Upon examining the cause of the generation of heterophase crystal grains gathered in the form of wrinkles during the manufacturing process of conventional technology, even upon performing hot forging and recrystallization annealing thereafter, primary crystal grains (roughly 50mm) remained in the ingot or billet, and

with a recrystallization temperature of roughly 1173K (900°C), it looks as though the recrystallized grains are merely generating in the primary crystal grains.

In other words, although it looks as though the primary crystal grains are crushed with the forging step and mostly eliminated, with the subsequent recrystallization temperature of roughly 1173K, the destruction of the primary crystals is incomplete, and it is considered that a part of this remains as traces of the primary crystal.

This is not eliminated even with the subsequent forging and recrystallization annealing steps, and, this is considered to become the heterophase crystal grains gathered in the form of wrinkles at the final stage of finish processing.

In light of the above, it is necessary to destroy the cast structure in the forging step, and to sufficiently perform recrystallization. Thus, in the present invention, a Ta ingot or billet formed by melting and casting is subject to forging, annealing, rolling processing and the like to prepare a sputtering target, wherein the ingot or billet is forged and thereafter subject to recrystallization annealing at a temperature of 1373K to 1673K.

As a result, it is possible to eliminate the generation of heterophase crystal grains gathered in the form of wrinkles in the Ta target, make the evenness (uniformity) of the film favorable, suppress the generation of arcing and particles, and improve the quality of the sputtered deposition.

As the standard manufacturing method of the present invention, for instance, a tantalum raw material (purity of 4N5 or more) is subject to electronic beam melting, thereafter cast to prepare an ingot or billet, this is subsequently subject to cold forging – recrystallization annealing at a temperature of 1373K to 1673K – cold forging – recrystallization annealing at a temperature of 1373K to 1673K – cold forging – recrystallization annealing at a temperature between the recrystallization starting temperature and 1373K – cold (hot) rolling – recrystallization annealing at a temperature between the recrystallization starting temperature and 1373K – finish processing to form a target material.

In the foregoing process, although the recrystallization annealing step at a temperature of 1373K to 1673K may be performed only once, by repeating this twice, the wrinkle shaped defects can be effectively reduced. With the temperature being lower than 1373K, it is difficult to eliminate the foregoing wrinkle shaped defects, and, with the temperature being higher than 1673K, abnormal grain

growth will occur and the grain diameter will become uneven. Thus, it is desirable to set the temperature to be 1673K or lower.

After eliminating the foregoing wrinkle shaped defects by performing recrystallization annealing at a temperature of 1373K to 1673K, the recrystallization annealing process after forging or rolling may be performed at a temperature between the recrystallization starting temperature and 1373K.

After the final rolling process, recrystallization annealing is performed at a temperature between the recrystallization starting temperature and 1373K, and finish processing (machine processing or the like) is performed thereto to form a target shape.

As a result of performing the foregoing processes, the wrinkle shaped defects of the Ta target can be eliminated, and a Ta target superior in uniformity having fine crystal grains in which the average crystal grain diameter thereof is 80 μm or less, and even 30 to 60 μm.

Examples and Comparative Examples

The present invention is now explained in detail with reference to the Examples. These Examples are merely illustrative, and the present invention shall in no way be limited thereby. In other words, the present invention shall only be limited by the scope of claim for a patent, and shall include the various modifications other than the Examples of this invention.

(Example 1)

A tantalum raw material having a purity of 99.997% was subject to electron beam melting, and this was cast to prepare an ingot or billet having a thickness of 200mm and diameter of 200mmϕ. The crystal grain diameter in this case was approximately 55mm. Next, after rolling this ingot or billet at room temperature, this was subject to recrystallization annealing at a temperature of 1500K. As a result, a material having a structure in which the average crystal grain diameter is 200 ìm, thickness of 100mm, and diameter of 100mmϕ was obtained.

Next, this was subject to cold mix forging at room temperature once again, and recrystallization annealing was performed thereto again at a temperature of 1480K. As a result, a material having a structure in which the average crystal grain diameter is 100μm, thickness of 100mm, and diameter of 100mmϕ was

obtained.

Next, this was subject to cold mix forging and recrystallization annealing at 1173K, subsequently subject to cold rolling and recrystallization annealing at 1173K once again, as well as finish processing, so as to obtain a target material having a thickness of 10mm and diameter of 320mmϕ.

As a result of performing the foregoing process, it was possible to obtain a Ta target superior in uniformity without any wrinkle shaped defects, and having fine crystal grains in which the average crystal grain diameter thereof is 60 μm. Further, the micrograph of this Ta target obtained in Example 1 had the same crystal structure as the Ta target shown in Fig. 1.

Upon performing sputtering with this Ta target, it was possible to improve the quality of the sputtered deposition in which the evenness (uniformity) of the film is favorable, the film thickness variation being 5% in an 8-inch wafer, and without any generation of arcing or particles.

(Example 2)

A tantalum raw material having a purity of 99.997% was subject to electron beam melting, and this was cast to prepare an ingot or billet having a thickness of 200mm and diameter of 200mmϕ. The crystal grain diameter in this case was approximately 50mm. Next, performing cold extend forging to this ingot or billet at room temperature, this was subject to recrystallization annealing at a temperature of 1500K. As a result, a material having a structure in which the average crystal grain diameter is 200μm, thickness of 100mm, and diameter of 100mmϕ was obtained.

Next, this was subject to cold mix forging at room temperature once again, and recrystallization annealing was performed thereto again at a temperature of 1173K. As a result, a material having a structure in which the average crystal grain diameter is 80μm, thickness of 100mm, and diameter of 100mmϕ was obtained.

Next, this was subject to cold mix forging and recrystallization annealing at 1173K, subsequently subject to cold rolling and recrystallization annealing at 1173K once again, as well as finish processing, so as to obtain a target material having a thickness of 10mm and diameter of 320mmϕ.

As a result of performing the foregoing process, it was possible to obtain a Ta

target superior in uniformity without any wrinkle shaped defects, and having fine crystal grains in which the average crystal grain diameter thereof is 35 μm. Further, the micrograph of this Ta target obtained in Example 2 had the same crystal structure as the Ta target shown in Fig. 1.

Upon performing sputtering with this Ta target, it was possible to improve the quality of the sputtered deposition in which the evenness (uniformity) of the film is favorable, the film thickness variation being 5% in an 8-inch wafer, and without any generation of arcing or particles.

(Example 3)

A tantalum raw material having a purity of 99.997% was subject to electron beam melting, and this was cast to prepare an ingot or billet having a thickness of 200mm and diameter of 300mmϕ. The crystal grain diameter in this case was approximately 50mm. Next, after performing cold extend forging to this ingot or billet at room temperature, this was subject to recrystallization annealing at a temperature of 1500K. As a result, a material having a structure in which the average crystal grain diameter is 250 μm, thickness of 100mm, and diameter of 100mmϕ was obtained.

Next, this was subject to cold mix forging at room temperature once again, and recrystallization annealing was performed thereto again at a temperature of 1173K. As a result, a material having a structure in which the average crystal grain diameter is 80 μm, thickness of 100mm, and diameter of 100mmϕ was obtained.

Next, this was subject to cold mix forging and recrystallization annealing at 1173K, subsequently subject to cold rolling and recrystallization annealing at 1173K once again, as well as finish processing, so as to obtain a target material having a thickness of 10mm and diameter of 320mmϕ.

As a result of performing the foregoing process, it was possible to obtain a Ta target superior in uniformity without any wrinkle shaped defects, and having fine crystal grains in which the average crystal grain diameter thereof is 50 μm. Further, the micrograph of this Ta target obtained in Example 3 had the same crystal structure as the Ta target shown in Fig. 1.

Upon performing sputtering with this Ta target, it was possible to improve the quality of the sputtered deposition in which the evenness (uniformity) of the film is

favorable, the film thickness variation being 6% in an 8-inch wafer, and without any generation of arcing or particles.

(Comparative Example 1)

As with Example 1, a tantalum raw material having a purity of 99.997% was subject to electron beam melting, and this was cast to prepare an ingot or billet having a thickness of 200mm and diameter of 200mm ϕ. The crystal grain diameter in this case was approximately 55mm. Next, after performing cold mix forging to this ingot or billet at room temperature, this was subject to recrystallization annealing at a temperature of 1173K. As a result, a material having a structure in which the average crystal grain diameter is 180 μm, thickness of 100mm, and diameter of 100mm ϕ was obtained.

Next, this was subject to cold mix forging at room temperature once again, and recrystallization annealing was performed thereto again at a temperature of 1173K. As a result, a material having a structure in which the average crystal grain diameter is 80 μm, thickness of 100mm, and diameter of 100mm ϕ was obtained.

Next, this was subject to cold rolling and recrystallization annealing at 1173K, as well as finish processing, so as to obtain a target material having a thickness of 10mm and diameter of 320mm ϕ.

Numerous traces in the form of wrinkles were observed from the center to the periphery of the Ta target obtained with the foregoing process, and the result was a Ta target having a heterophase crystal structure. Further, the micrograph of the Ta target obtained in Comparative Example 1 had the same crystal structure as the Ta target shown in Fig. 3.

Upon performing sputtering with this Ta target, it deteriorated the quality of the sputtered deposition in which the evenness (uniformity) of the film is inferior, the film thickness variation being 10% in an 8-inch wafer, and with the generation of arcing or particles.

Effect of the Invention

The present invention yields a superior effect in the manufacturing method of a Ta sputtering target as a result of adjusting the crystal grains by performing forging, recrystallization annealing, rolling processing and the like to the ingot or billet

material, eliminating the generation of heterophase crystal grains gathered in the form of wrinkles in the target, making the evenness (uniformity) of the film favorable, suppressing the generation of arcing and particles, and improving the quality of the sputtered deposition.